



Europäisches Patentamt
European Patent Office
Office européen des brevets



(11) Publication number:

0 359 362 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication of patent specification: 05.04.95 (51) Int. Cl.⁶: **C09C 3/10, C09C 3/06**

(21) Application number: 89306595.3

(22) Date of filing: 29.06.89

(54) **Process for providing an inorganic filler with a coating containing a latex and carbon black, obtained product and its use.**

(30) Priority: 13.07.88 GB 8816622
13.12.88 US 283564

(43) Date of publication of application:
21.03.90 Bulletin 90/12

(45) Publication of the grant of the patent:
05.04.95 Bulletin 95/14

(84) Designated Contracting States:
AT BE CH DE ES FR GR IT LI LU NL SE

(56) References cited:
EP-A- 0 277 244
FR-A- 2 338 978
GB-A- 2 179 665

(73) Proprietor: **ECC INTERNATIONAL LIMITED**
1015 Arlington Business Park
Theale
Reading
Berkshire RG7 4SA (GB)

(72) Inventor: **Jeffs, David George**
Brambletye
Garker Road
Trethurgy
St Austell
Cornwall PL 25 4DJ (GB)

(74) Representative: **Bull, Michael Alan et al**
Haseltine Lake & Co.
Hazlitt House
28 Southampton Buildings
Chancery Lane
London WC2A 1AT (GB)

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid (Art. 99(1) European patent convention).

EP 0 359 362 B1

Description

This invention relates to inorganic fillers for organic polymer compositions, to a process for preparing such fillers, to organic polymer compositions including such fillers and to articles made from such organic polymer compositions.

In a known process for making glass mat reinforced thermoplastic sheet material a mixture of chopped glass fibres, polypropylene powder and an inorganic filler such as kaolin, calcium carbonate, talc or mica is dispersed in water containing an anionic surfactant using a high speed mixer to form a stable foam and this foam is spread uniformly over the surface of a travelling wire mesh felt similar to that conventionally used in a Fourdrinier paper making machine and the aqueous phase is withdrawn through the wire mesh by gravity and suction. The mat of solid materials thus formed on the upper surface of the wire mesh is then dried at a temperature of about 100°C and the resultant dried mat may be cut into pieces of suitable size, stacked if desired to form a multi-layer sheet material and pressed between steel plates at a temperature of about 200°C and allowed to cool in the press to form a sheet-like component such as a car body panel. Alternatively, the stacks of cut pieces may be heated to about 200 °C by means of a current of hot air or by infra red radiation and subsequently allowed to cool in a press at about 60 °C to form the sheet-like material.

In addition to the glass fibres, polypropylene and inorganic filler it is often necessary to include in the starting mixture for a glass mat reinforced thermoplastic material a small amount, normally up to about 4% by weight of the total solid material, of carbon black. This is introduced not only to render the final sheet material black in colour, as is often required in commerce, but also to provide a screening effect against the harmful action of ultraviolet radiation on the material. However, when carbon black is introduced into the starting mixture, problems are experienced because of the well known difficulties of handling fine carbon black powder without creating a cloud of black particles which settles out over a wide area. This problem is especially severe when dry carbon black is mixed with a dry thermoplastic material. The retention of the fine carbon black particles in the mat during the forming operation on the wire mesh has also been found to be poor and it is generally found that the carbon black is unevenly dispersed in the final dried and pressed material with the result that the sheet material has a veined or mottled appearance with dark areas having a high concentration of carbon black alternating with areas which are light in colour and almost translucent.

Similar handling problems arise when fine carbon black is incorporated in injection moulding thermoplastic compositions in which carbon black in an amount of about 1% by weight of the total composition is normally required, and in thermosetting dough moulding compounds in which the amount of carbon black is usually about 0.5% by weight based on the total weight of the compound.

Carbon black is the term used to describe a wide range of fine carbon products which may be made by partial combustion or thermal decomposition of hydrocarbons in the vapour phase. The ultimate colloidal units of carbon black may occur as aggregates, which are fused assemblies of particles. Typically, carbon blacks may have particle diameters up to 500 nanometers. Most carbon blacks have an elemental carbon composition greater than 90%.

GB-A-2179665 describes a process for preparing an inorganic filler which process comprises treating a particulate inorganic material with a natural or synthetic latex composition and dewatering and drying the resultant mixture. The surface-treated inorganic filler can be used with advantage in a polymeric resin composition.

US-A-4366285 relates to a process for producing an elastomer-filler mixture optionally containing an extender oil. The composite material contains from 20 to 70% by weight of filler based on the weight of the elastomer. In some of the composite materials, a high-aromatic extender oil and a finely divided carbon black are included.

EP-0213931 describes a process for preparing an inorganic filler for use in a polymeric resin composition.

The present invention provides a method of incorporating finely divided carbon black pigment into a thermoplastic or thermosetting organic polymer composition in such a way that the handling problems are overcome and the carbon black is substantially uniformly dispersed in the polymer composition.

According to one aspect of the present invention, there is provided a process for preparing a coated inorganic filler which comprises:

- (1) combining a particulate inorganic material, a natural or synthetic latex composition and a dispersed aqueous suspension of carbon black to form an aqueous mixture of the ingredients; and
- (2) dewatering and drying the resultant mixture;

said natural or synthetic latex composition being employed in an amount such as to provide a coating on the particles of inorganic filler of from 1% to 10% by weight of latex solids based on the weight of dry

inorganic material.

According to another aspect of the present invention, there is provided a coated particulate inorganic material comprising a natural or synthetic elastomer and carbon black, wherein the elastomer is present in the coating in an amount of from 1% to 10% by weight, based on the weight of inorganic material.

5 The particulate inorganic material may be combined or admixed with the latex composition and the dispersed aqueous suspension of carbon black sequentially or simultaneously. Admixing may be sequential with the latex composition first being combined in aqueous suspension with the particulate inorganic material to which the dispersed aqueous suspension of the carbon black is then added. Alternatively, and
10 presently preferred, is first to combine the dispersed aqueous suspension of carbon black with an aqueous suspension of the particulate inorganic material to form a mixed suspension and then to add the latex composition. The resultant mixture of carbon black, inorganic filler and latex may be dewatered and dried by spray drying to form hollow dry microspheres.

The inorganic material may be chosen from a kaolinitic clay (e.g. kaolin or ball clay), a calcined kaolinitic clay, calcium carbonates, silicates of aluminium and calcium (e.g. the natural calcium silicate
15 known as wollastonite), bauxite, talc, mica, alumina trihydrate, silica, carbonates and hydroxides of magnesium (e.g. natural hydrotalcite), dolomite (i.e. the natural double carbonate of calcium and magnesium), calcium sulphate (e.g. gypsum), and titanium dioxide. The inorganic material may be natural or synthetic and, in particular, both natural and synthetic forms of calcium carbonate, silicates of aluminium and calcium, silica, carbonates and hydroxides of magnesium, calcium sulphate and titanium dioxide are
20 within the scope of this invention. Where the material is synthetic it may be precipitated (as with calcium carbonate, silica and titanium dioxide). The inorganic materials specified above may be regarded as white inorganic materials; the term "white" used in relation to "inorganic material" does not mean, however, that the mineral necessarily has a pure white colour, but that it is substantially free of any strong non-white hue. Many of the inorganic materials which may be employed in the present invention are crystalline.

25 Preferably, the particles should be no larger than 100 microns, more preferably 50 microns and most preferably 20 microns, in diameter.

Thus, the invention enables carbon black to be incorporated into an organic polymer composition as a component of a coating on an inorganic filler material, the carbon black being held in the coating by a film of a natural or synthetic elastomer material. The three main components (carbon black, inorganic filler and
30 latex) are dispersed together in water preferably containing a dispersing agent, and dewatered and dried, for example by spray drying to form dry microspheres.

Preferably, the process of the invention comprises the following steps:

- a) fine carbon black is mixed with water containing a dispersing agent to form a suspension preferably containing from 5% to 25% by weight of carbon black;
- 35 b) a suspension preferably containing from 50% to 65% on a dry weight basis of a white inorganic filler and a dispersing agent for the inorganic filler is mixed with the carbon black suspension formed in step a) in proportions such that the quantity of carbon black is preferably from 0.1% to 10% on a dry weight basis based on the weight of the inorganic filler;
- c) there is mixed with the inorganic filler/carbon black suspension formed in step b) sufficient of a natural
40 or synthetic latex composition to provide from 1% to 10% by weight of latex solids, based on the weight of the inorganic filler; and
- d) the mixed suspension formed in step c) is spray dried in a spray dryer to form hollow microspheres of substantially dry material having diameters preferably in the range from 0.02 mm to 0.5 mm.

In step a) the dispersing agent may be, for example, a water-soluble salt of poly(acrylic acid), poly-
45 (methacrylic acid) or of a copolymer containing from 10% to 80% by weight of acrylonitrile or methacrylonitrile monomer units and from 90% to 20% by weight of acrylic acid or methacrylic acid monomer units. The number average molecular weight of the water soluble polymer is preferably not more than 10,000. The amount of the dispersing agent used is preferably in the range from 0.1% to 5% by weight, based on the weight of dry carbon black.

50 In step b) the inorganic filler may be a white inorganic material as listed above. The dispersing agent for the inorganic filler is advantageously a water soluble salt of poly(acrylic acid) or poly(methacrylic acid) having a number average molecular weight of not more than 10,000. The inorganic filler does not require any chemical pretreatment other than dispersion with a dispersing agent. The amount of the dispersing agent used is preferably in the range of from 0.05% to 0.5% by weight, based on the weight of the dry
55 inorganic filler.

The latex may be of a natural rubber, or of a natural rubber which has been substituted with functional groups, or a synthetic rubber such as a styrene butadiene rubber (SBR). Other suitable latices include those formed from acrylic copolymers (either elastomeric or non-elastomeric, although elastomeric is preferred)

and non-elastomeric materials such as poly(vinyl acetate) and copolymers of vinyl acetate. The latex composition, which is a stabilised suspension of polymer particles in water, typically contains 40% to 60% by weight of solids. The latex may be stabilised with the aid of a surfactant or a water-soluble colloid although a surfactant would normally be employed as it usually gives a latex of lower viscosity.

5 The acrylic copolymer may be a copolymer of a lower alkyl ester of acrylic acid with a lower alkyl ester of methacrylic acid. Particularly preferred are copolymers of ethyl acrylate and methyl methacrylate. Also suitable are copolymers of one or other or both of a lower alkyl ester of acrylic acid and a lower alkyl ester of methacrylic acid with a further monomer chosen from vinyl acetate, styrene, acrylonitrile and mixtures thereof. The alkyl chains of the lower alkyl esters of acrylic acid and methacrylic acid preferably have from
10 one to four carbon atoms.

The copolymers of vinyl acetate may be those formed by copolymerising vinyl acetate with a copolymerising monomer chosen from a lower alkyl ester of acrylic acid, a lower alkyl ester of methacrylic acid, styrene, acrylonitrile and mixtures thereof.

The mixed suspension formed in step c) may also contain other ingredients which are required in
15 certain organic polymer compositions such as antioxidants and metal deactivators. For example, an antioxidant, which may be, for example, of the non-staining hindered phenol type, may be mixed with water to form a suspension containing from 40% by weight to 60% by weight of the dry antioxidant, and the suspension mixed with the suspension formed in step c) in proportions such as to provide from 0.1% to 10% by weight of the dry antioxidant based on the weight of the dry inorganic filler.

20 In step d) the inlet temperature of the spray dryer is preferably less than 400°C, and most preferably less than 350°C, in order to avoid thermal degradation of the elastomer material or combustion of the carbon black.

Coated inorganic material in accordance with the present invention may be incorporated into organic polymer compositions from which articles may be partially, or wholly, formed. The organic polymer
25 composition into which the filler may be incorporated can be in a solid (i.e. powder) or liquid (i.e. resin) form. The coated inorganic filler of the present invention is particularly suited to organic polymer compositions in powder form.

The dry hollow microspheres prepared by the process of the present invention are found to be in the form of a free-flowing powder which can be easily incorporated into an organic polymer composition and
30 the carbon black is so well bound into the microspheres that the material may be poured onto a sheet of white paper with at most only a trace of black staining being visible after removal of the microspheres. The microspheres may, by adjustment of the gas flow rate through the spray dryer and of the inlet temperature, be prepared of such a size that the retention of the microspheres in, for example, a glass mat reinforced thermoplastic composition is greatly improved. Most advantageously the microspheres should have diam-
35 eters in the range from 0.3 mm to 0.5 mm.

In the present invention, any commercial grade of carbon black is suitable; however, fine carbon black, i.e. that having an average particle diameter no greater than 300 nanometers is particularly preferred.

The dry microspheres prepared in accordance with the invention have also been found to be very suitable for incorporation into organic polymer compositions such as injection moulding thermoplastic
40 compositions and dough moulding thermosetting compositions.

The invention is illustrated by the following Examples.

EXAMPLE 1

45 A carbon black powder having an average particle diameter of 0.02 micrometer was dispersed in a quantity of water so as to form a suspension containing 20% by weight of the carbon black, there being dissolved in the water 2% by weight, based on the weight of the dry carbon black of a sodium polyacrylate dispersing agent having a number average molecular weight of 1680. The carbon black suspension was then added to a suspension which had been formed by mixing a suspension containing 60% by weight of a
50 paper filler grade kaolin clay and 0.2% by weight, based on the weight of dry kaolin, of the same dispersing agent as was used for the carbon black, and a latex containing 50% by weight of an elastomeric acrylic copolymer of ethyl acrylate and methyl methacrylate in proportions such that the quantity of the dry elastomeric copolymer was 5% by weight, based on the weight of dry kaolin clay. The kaolin clay had a particle size distribution such that 20% by weight consisted of particles having an equivalent spherical
55 diameter larger than 10 microns and 35% by weight consisted of particles having an equivalent spherical diameter smaller than 2 microns.

The carbon black suspension was mixed with the kaolin/elastomeric copolymer suspension in proportions such that the quantity of carbon black was 5% by weight, based on the weight of dry kaolin clay.

The resultant mixed suspension was fed to the atomizer of a spray dryer the inlet temperature of which was 300°C and the solid components of the mixture were recovered in the form of dry hollow microspheres of diameters in the range from 0.3 to 0.5 mm.

5 EXAMPLE 2

A glass mat reinforced thermoplastic composition was prepared according to the following formulation:-

10	Chopped glass fibres	40g
	Polypropylene powder (0.3 mm - 0.5 mm diameter)	110g
	Composite filler particles prepared in Example 1	50g

The ingredients were mixed into 7 litres of water containing 20ml of an anionic surfactant using a high speed mixer to form a stable foam. The foam was distributed evenly over a wire mesh screen and the water removed by gravity and suction. The resultant felted mat was dried at 100°C and then pressed at 200°C between steel plates and allowed to cool in the press. the resultant glass mat reinforced polypropylene sheet was a uniform deep black in colour with no visible streaks, veins or mottling. The retention of the filler particles in the mat was also extremely good.

20 EXAMPLE 3

A further sample of the same carbon black powder as was used in Example 1 was dispersed in water containing 2% by weight, based on the weight of the dry carbon black, of the same dispersing agent as was used in Example 1 to form a suspension containing 20% by weight of the carbon black.

This suspension was then added to a suspension containing 60% by weight of a ground natural mica, having a particle size distribution such that substantially all of the particles had an equivalent spherical diameter smaller than 10 microns, and 0.2% by weight, based on the weight of dry mica, of the same dispersing agent as was used for the carbon black. To the mica/carbon black suspension thus formed, there was then added firstly a latex containing 50% by weight of the same acrylic copolymer as was used in Example 1 in proportions such that the quantity of the dry elastomeric copolymer was 5% by weight, based on the weight of dry mica, and secondly a suspension containing 50% by weight of a non-staining antioxidant of the hindered phenol type in proportions such that the quantity of the dry antioxidant was 1% by weight, based on the weight of dry mica. The quantity of carbon black in the resultant mixed suspension was 5% by weight, based on the weight of dry mica.

The resultant mixed suspension was spray dried under the same conditions as were described in Example 1 and the solid components of the mixture were recovered in the form of dry hollow microspheres of diameters in the range from 0.1 to 0.5 mm.

These microspheres were incorporated into a glass mat reinforced thermoplastic composition following the same formulation and method as were described in Example 2. The resultant glass mat reinforced polypropylene sheet was a uniform black in colour with no visible streaks, veins or mottling. The retention of the filler particles in the mat was also extremely good.

40 EXAMPLE 4

The experiment of Example 3 was repeated except that the mica was replaced by an equal quantity of a synthetic alumina trihydrate having a particle size distribution such that substantially all the particles had an equivalent spherical diameter smaller than 10 microns.

Again the resultant glass mat reinforced polypropylene sheet was a uniform black in colour with no visible streaks, veins or mottling. The retention of the filler particles in the mat was extremely good.

Claims

1. A process for preparing a coated inorganic filler which comprises:
 - (1) combining a particulate inorganic material, a natural or synthetic latex composition and a dispersed aqueous suspension of carbon black to form an aqueous mixture of the ingredients; and
 - (2) dewatering and drying the resultant mixture;
 said natural or synthetic latex composition being employed in an amount such as to provide a

coating on the particles of inorganic filler of from 1% to 10% by weight of latex solids based on the weight of dry inorganic material.

2. A process according to claim 1, wherein the carbon black is employed in an amount such as to provide in the coating from 0.1% to 10% by weight of dry carbon black based on the weight of the inorganic filler.
3. A process according to claim 1 or 2, wherein the inorganic material and the latex composition are added, either sequentially or simultaneously, to the aqueous suspension of carbon black.
4. A process according to claim 1, 2 or 3, wherein the aqueous suspension of carbon black contains from 5% to 25% by weight of carbon black.
5. A process according to any preceding claim, wherein the particulate inorganic material is employed in the form of a dispersed aqueous suspension.
6. A process according to claim 5, wherein the aqueous suspension of the inorganic material contains from 50% to 65% by weight of the inorganic material.
7. A process according to any preceding claim, wherein the mixture of inorganic material, latex and carbon black is spray dried to form hollow microspheres.
8. A process according to any preceding claim, wherein the particulate inorganic material is chosen from a kaolinitic clay, a calcined kaolinitic clay, a calcium carbonate, silicates of calcium and aluminium, bauxite, talc, mica, alumina trihydrate, silica, carbonates and hydroxides of magnesium, dolomite, calcium sulphate and titanium dioxide.
9. A process according to any preceding claim, wherein the latex solids are chosen from a natural rubber, a natural rubber which has been substituted with functional groups, a synthetic rubber, an acrylic copolymer, a poly(vinyl acetate) and copolymers of vinyl acetate.
10. A process according to claim 1, wherein:
the particulate inorganic material is chosen from a kaolinitic clay, a calcined kaolinitic clay, a calcium carbonate, silicates of calcium and aluminium, bauxite, talc, mica, alumina trihydrate, silica, carbonates and hydroxides of magnesium, dolomite, calcium sulphate or titanium dioxide; and wherein:
the natural or synthetic latex composition comprises a suspension of polymer particles in water wherein the latex solids are chosen from a natural rubber, a natural rubber which has been substituted with functional groups, a synthetic rubber, an acrylic copolymer, a poly(vinyl acetate) and a copolymer of vinyl acetate.
11. A process according to claim 1, wherein: step (1) is effected by:
(a) forming a dispersed aqueous suspension of fine carbon black which contains from 5% to 25% by weight, on a dry weight basis, of the carbon black;
(b) forming a dispersed aqueous suspension of a white inorganic material containing from 50% to 60% by weight on a dry weight basis, of the inorganic material;
(c) combining the aqueous suspension of the inorganic material with the natural or synthetic latex composition; and
(d) combining the dispersed aqueous suspension of carbon black with the inorganic mineral/latex suspension formed in step (c) in proportions such that the quantity of carbon black is from 0.1% to 10% on a dry weight basis, based on the weight of the inorganic mineral; and wherein:
step (2) is effected by:
(e) spray drying the suspension formed in step (d) to form hollow microspheres of substantially dry material having diameters in the range of from 0.02 mm to 0.5 mm.
12. A process according to claim 1, wherein:
step (1) is effected by:
(a) forming a dispersed aqueous suspension of fine carbon black which contains from 5% to 25% by weight, on a dry weight basis, of the carbon black;

- (b) forming a dispersed aqueous suspension of a white inorganic material containing from 50% to 60% by weight on a dry weight basis, of the inorganic material;
- (c) combining the aqueous suspension of the inorganic material with the dispersed aqueous suspension of carbon black formed in step (a) in proportions such that the quantity of carbon black is from 0.1% to 10% on a dry weight basis, based on the weight of the inorganic mineral; and
- (d) combining the inorganic mineral/carbon black formed in step (c) with the natural or synthetic latex composition; and wherein:
- step (2) is effected by:
- (e) spray drying the suspension formed in step (d) to form hollow microspheres of substantially dry material having diameters in the range of from 0.02 mm to 0.5 mm.

- 13. An organic polymer composition which comprises an organic polymeric material and an inorganic filler as prepared by a process as claimed in any one of claims 1 to 12.
- 14. An organic polymer composition as claimed in claim 13, wherein the polymeric material is a thermoplastic or a thermosetting material.
- 15. An article formed, either partially or wholly, from an organic polymer composition as claimed in claim 13 or 14.

Patentansprüche

- 1. Verfahren zur Herstellung eines beschichteten anorganischen Füllstoffs, umfassend:
 - (1) Vereinigen eines partikelförmigen anorganischen Materials, einer natürlichen oder synthetischen Latexzusammensetzung und einer dispergierten wäßrigen Suspension von Kohlenstoffschwarz, wodurch eine wäßrige Mischung der Bestandteile hergestellt wird; und
 - (2) Entwässern und Trocknen der entstandenen Mischung;
 - wobei die natürliche oder synthetische Latexzusammensetzung in einer Menge angewendet wird, daß auf den Partikeln des anorganischen Füllstoffs eine Beschichtung von 1 bis 10 Gew.% Latexfeststoffe auf der Basis des trockenen anorganischen Materials gebildet wird.
- 2. Verfahren nach Anspruch 1, wobei Kohlenstoffschwarz in einer Menge verwendet wird, daß eine Beschichtung von 0,1 bis 10 Gew.% trockenes Kohlenstoffschwarz auf der Basis des anorganischen Füllstoffs gebildet wird.
- 3. Verfahren nach Anspruch 1 oder 2, wobei das anorganische Material und die Latexzusammensetzung der wäßrigen Suspension von Kohlenstoffschwarz entweder nacheinander oder gleichzeitig zugesetzt werden.
- 4. Verfahren nach Anspruch 1, 2 oder 3, wobei die wäßrige Suspension von Kohlenstoffschwarz 5 bis 25 Gew.% Kohlenstoffschwarz enthält.
- 5. Verfahren nach einem der vorstehenden Ansprüche, wobei das partikelförmige anorganische Material in Form einer dispergierten wäßrigen Suspension verwendet wird.
- 6. Verfahren nach Anspruch 5, wobei die wäßrige Suspension des anorganischen Materials 50 bis 65 Gew.% des anorganischen Materials enthält.
- 7. Verfahren nach einem der vorstehenden Ansprüche, wobei die Mischung aus anorganischem Material, Latex und Kohlenstoffschwarz zu Hohlperlen sprühgetrocknet wird.
- 8. Verfahren nach einem der vorstehenden Ansprüche, wobei das partikelförmige anorganische Material aus Kaoliniton, gebranntem Kaoliniton, Calciumcarbonat, Silicaten von Calcium und Aluminium, Bauxit, Talkum, Glimmer, Aluminiumoxidtrihydrat, Siliciumdioxid, Carbonaten und Hydroxiden von Magnesium, Dolomit, Calciumsulfat und Titandioxid ausgewählt ist.
- 9. Verfahren nach einem der vorstehenden Ansprüche, wobei die Latexfeststoffe aus natürlichem Kautschuk, mit funktionellen Gruppen substituiertem natürlichem Kautschuk, synthetischem Kautschuk, einem

Acrylcopolymer, Poly(vinylacetat) und Copolymeren von Vinylacetat ausgewählt sind.

10. Verfahren nach Anspruch 1, wobei:

das partikelförmige anorganische Material aus Kaolinitton, gebranntem Kaolinitton, Calciumcarbonat, Silicaten von Calcium und Aluminium, Bauxit, Talkum, Glimmer, Aluminiumoxidtrihydrat, Siliciumdioxid, Carbonaten und Hydroxiden von Magnesium, Dolomit, Calciumsulfat oder Titandioxid ausgewählt ist; und

die natürliche oder synthetische Latexzusammensetzung eine Suspension von Polymerpartikeln in Wasser umfaßt, wobei die Latexfeststoffe aus natürlichem Kautschuk, mit funktionellen Gruppen substituiertem natürlichem Kautschuk, synthetischem Kautschuk, einem Acrylcopolymer, Poly(vinylacetat) und einem Copolymer von Vinylacetat ausgewählt sind.

11. Verfahren nach Anspruch 1, wobei:

der Schritt (1) erfolgt:

(a) Herstellen einer dispergierten wäßrigen Suspension von feinem Kohlenstoffschwarz, die auf Trockengewichtsbasis 5 bis 25 Gew.% Kohlenstoffschwarz enthält;

(b) Herstellen einer dispergierten wäßrigen Suspension eines weißen anorganischen Materials, die auf Trockengewichtsbasis 50 bis 60 Gew.% des anorganischen Materials enthält;

(c) Vereinigen der wäßrigen Suspension des anorganischen Materials mit der natürlichen oder synthetischen Latexzusammensetzung; und

(d) Vereinigen der dispergierten wäßrigen Suspension von Kohlenstoffschwarz mit der im Schritt (c) hergestellten Suspension aus anorganischem Material/Latex in Anteilen, so daß die Menge an Kohlenstoffschwarz auf Trockengewichtsbasis 0,1 bis 10% auf der Basis des Gewichtes des anorganischen Materials beträgt; und

der Schritt (2) erfolgt:

(e) Sprühtrocknen der im Schritt (d) hergestellten Suspension, wodurch Hohlperlen des im wesentlichen trockenen Materials mit Durchmessern im Bereich von 0,02 bis 0,5 mm gebildet werden.

12. Verfahren nach Anspruch 1, wobei:

der Schritt (1) erfolgt:

(a) Herstellen einer dispergierten wäßrigen Suspension von feinem Kohlenstoffschwarz, die auf Trockengewichtsbasis 5 bis 25 Gew.% Kohlenstoffschwarz enthält;

(b) Herstellen einer dispergierten wäßrigen Suspension eines weißen anorganischen Materials, das auf Trockengewichtsbasis 50 bis 60 Gew.% des anorganischen Materials enthält;

(c) Vereinigen der wäßrigen Suspension des anorganischen Materials mit der im Schritt (a) hergestellten dispergierten wäßrigen Suspension von Kohlenstoffschwarz in Anteilen, so daß die Menge an Kohlenstoffschwarz auf Trockengewichtsbasis 0,1 bis 10% auf der Basis des Gewichtes des anorganischen Materials beträgt; und

(d) Vereinigen des im Schritt (c) hergestellten anorganischen Materials/Kohlenstoffschwarz mit der natürlichen oder synthetischen Latexzusammensetzung; und

der Schritt (2) erfolgt:

(e) Sprühtrocknen der im Schritt (d) hergestellten Suspension, wodurch Hohlperlen des im wesentlichen trockenen Materials mit Durchmessern im Bereich von 0,02 bis 0,5 mm gebildet werden.

13. Organische Polymerzusammensetzung, die ein organisches Polymermaterial und einen anorganischen Füllstoff nach einem Verfahren nach einem der Ansprüche 1 bis 12 umfaßt.

14. Organische Polymerzusammensetzung nach Anspruch 13, wobei das Polymermaterial ein thermoplastisches oder duroplastisches Material ist.

15. Gegenstand, der entweder teilweise oder vollständig aus einer organischen Polymerzusammensetzung nach Anspruch 13 oder 14 hergestellt ist.

Revendications

55

1. Procédé de préparation d'une charge inorganique revêtue, selon lequel :

(1) on associe une matière inorganique particulière, une composition de latex naturelle ou synthétique, et une suspension aqueuse dispersée de noir de carbone, pour former un mélange aqueux des

ingrédients ; et

(2) on égoutte et on sèche le mélange résultant ; cette composition de latex naturelle ou synthétique étant employée selon une quantité appropriée pour former un revêtement sur les particules de charge inorganique, à raison de 1 % à 10 % en poids de solides de latex par rapport au poids de matière inorganique sèche.

5

2. Procédé selon la revendication 1, dans lequel le noir de carbone est employé selon une quantité appropriée pour obtenir, dans le revêtement, de 0,1 % à 10 % en poids de noir de carbone sec, par rapport au poids de la charge inorganique.

10

3. Procédé selon la revendication 1 ou 2, dans lequel la matière inorganique et la composition de latex sont ajoutées successivement ou simultanément dans la suspension aqueuse de noir de carbone.

15

4. Procédé selon la revendication 1, 2 ou 3, dans lequel la suspension aqueuse de noir de carbone contient de 5% à 25% en poids de noir de carbone.

5. Procédé selon l'une quelconque des revendications précédentes, dans lequel la matière inorganique particulaire est employée sous la forme d'une suspension aqueuse dispersée.

20

6. Procédé selon la revendication 5, dans lequel la suspension aqueuse de la matière inorganique contient de 50 % à 65 % en poids de la matière inorganique.

25

7. Procédé selon l'une quelconque des revendications précédentes, dans lequel le mélange de matière inorganique, de latex et de noir de carbone, est séché par atomisation afin de former des microsphères creuses.

30

8. Procédé selon l'une quelconque des revendications précédentes, dans lequel la matière inorganique particulaire est choisie parmi une argile à base de kaolinite, une argile à base de kaolinite calcinée, du carbonate de calcium, des silicates de calcium et d'aluminium, de la bauxite, du talc, du mica, de l'alumine tri-hydratée, de la silice, des carbonates et des hydroxydes de magnésium, la dolomite, le sulfate de calcium et le dioxyde de titane.

35

9. Procédé selon l'une quelconque des revendications précédentes, dans lequel les solides de latex sont choisis parmi un caoutchouc naturel, un caoutchouc naturel ayant été substitué avec des groupes fonctionnels, un caoutchouc synthétique, un copolymère acrylique, un poly(acétate de vinyle) et des copolymères d'acétate de vinyle.

40

10. Procédé selon la revendication 1, dans lequel :

la matière inorganique particulaire est choisie parmi une argile à base de kaolinite, un carbonate de calcium, les silicates de calcium et d'aluminium, la bauxite, le talc, le mica, l'alumine tri-hydratée, la silice, les carbonates et les hydroxydes de magnésium, la dolomite, le sulfate de calcium et le dioxyde de titane ; et dans lequel :

45

la composition de latex naturelle ou synthétique comprend une suspension de particules de polymère dans de l'eau, les solides de latex étant choisis parmi un caoutchouc naturel, un caoutchouc naturel ayant été substitué avec des groupes fonctionnels, un caoutchouc synthétique, un copolymère acrylique, un poly (acétate de vinyle) et un copolymère d'acétate de vinyle.

50

11. Procédé selon la revendication 1, dans lequel l'étape (1) est effectuée :

a) en formant une suspension aqueuse dispersée de noir de carbone fin, contenant de 5 % à 25 % en poids à sec, du noir de carbone ;

b) en formant une suspension aqueuse dispersée d'une matière inorganique blanche contenant de 50 % à 60 % en poids à sec, de la matière inorganique ;

c) en associant la suspension aqueuse de la matière inorganique, avec la composition de latex naturelle ou synthétique ; et

55

d) en associant la suspension aqueuse dispersée de noir de carbone, avec la suspension de minéral inorganique et de latex formée dans l'étape (c) selon des proportions telles que la quantité de noir de carbone soit de 0,1 % à 10 % en poids à sec par rapport au poids du minéral inorganique ; et dans lequel, l'étape (2) est effectuée :

e) en séchant par atomisation la suspension formée dans l'étape (d) afin de former des microsphères creuses d'une matière à peu près sèche ayant un diamètre de 0,02 mm à 0,5 mm.

12. Procédé selon la revendication 1, dans lequel l'étape (1) est effectuée :

- 5 a) en formant une suspension aqueuse dispersée de noir de carbone fin, contenant de 5 % à 25 % en poids à sec, du noir de carbone ;
b) en formant une suspension aqueuse dispersée d'une matière inorganique blanche contenant de 50 % à 60 % en poids à sec, de la matière inorganique ;
10 c) en associant la suspension aqueuse de la matière inorganique, avec la suspension aqueuse de noir de carbone formée dans l'étape (a), selon des proportions telles que la quantité de noir de carbone, soit de 0,1 % à 10 % en poids à sec par rapport au poids du minéral inorganique ; et
d) en associant le mélange de minéral inorganique et de noir de carbone formé dans l'étape (c) avec la composition de latex naturelle et synthétique ; et dans lequel, l'étape (2) est effectuée :
15 e) en séchant par atomisation la suspension formée dans l'étape (d) afin de former des microsphères creuses d'une matière à peu près sèche ayant un diamètre de 0,02 mm à 0,5 mm.

13. Composition à base de polymère organique, comprenant une matière polymère organique et une charge inorganique préparée selon un procédé revendiqué dans l'une quelconque des revendications 1 à 12.

20

14. Composition à base de polymère organique selon la revendication 13, dans laquelle la matière polymère est une matière thermoplastique ou thermodurcissable.

15. Article formé partiellement ou totalement à partir d'une composition à base de polymère organique, selon la revendication 13 ou 14.

25

30

35

40

45

50

55